

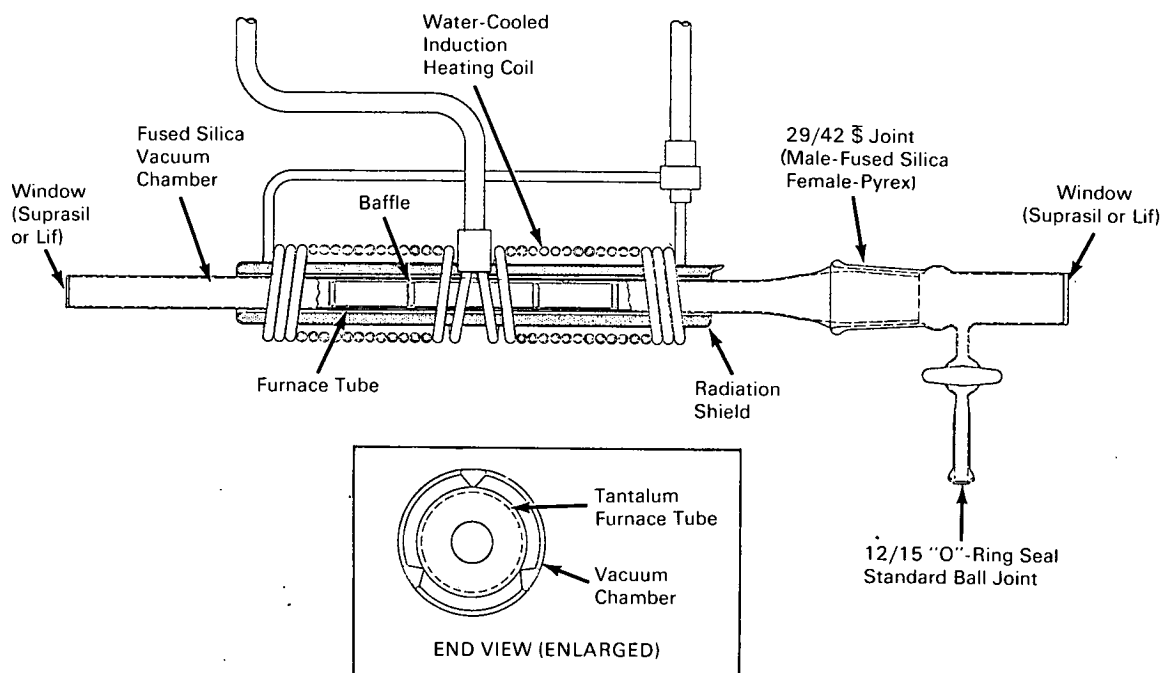


AEC-NASA TECH BRIEF



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Miniaturized King Furnace Permits Absorption Spectroscopy of Small Samples



The problem:

To devise an instrument for obtaining metal vapor absorption spectra of samples weighing only a few milligrams. Many research samples are handled in small quantities, either because of their scarcity or radioactivity. A King furnace has been a standard tool for producing absorption spectra of metal vapors. This furnace, however, is bulky, inconvenient to disassemble and reassemble for recharging, requires a large amount of power for heating, and requires a sample charge in the order of grams. There is also a problem of the sample reacting with the furnace tube material to form carbides.

The solution:

A miniature King-type furnace, consisting of an inductively heated, small diameter tantalum tube supported in a radiation shield, eliminates the disadvantages of the conventional furnace in obtaining absorption spectra of metal vapors. The modified furnace is compact, easily assembled, can be operated to a temperature of 1400°C, and gives well developed absorption spectra with sample charges of only a few milligrams of metal.

How it's done:

The furnace apparatus, shown in the diagram, consists of four basic parts: the furnace tube, the vacuum

(continued overleaf)

chamber, the radiation shield and the water-cooled induction heating coil. The furnace tube is a 14 cm length of 12.7 mm od seamless tantalum tubing with a 0.125 mm wall thickness. Four baffles, inserted to inhibit diffusion of the sample during operation, are formed by spinning 0.125 mm tantalum sheet over a suitable form. The baffle aperture is 4.75 mm.

The vacuum chamber is in two sections. One section, which carries the furnace tube, is a 34 cm length of fused silica tubing, with a male fused silica standard taper joint at one end. Thermal contact with the tantalum tube is minimized by supporting the latter on six small indentations in the silica tubing. This permits furnace operation well above the softening temperature of quartz without danger of collapsing the vacuum chamber. This chamber is completed by a female pyrex standard taper joint, a window, a vacuum connection made through a stop cock, an O-ring-seal, and a standard ball joint. The ends of the vacuum chamber are ground flat and the window attached with a low vapor pressure epoxy cement.

The radiation shield is a double-walled fused silica tube filled with finely divided carbon, thoroughly outgassed and sealed off under vacuum. The tubing forming the inner wall allows 0.2 mm clearance between it and the od of the vacuum chamber. After filling, a small plug of quartz wool is inserted through the pump-out connection and a constriction is formed in the tubing above this plug. The carbon powder is outgassed by sealing the assembly to a vacuum line, heating it in a tube furnace to 1050°C, and pumping to a vacuum of 10^{-5} Torr or higher. The outgassing takes about 24 hours and the shield is then sealed off under this high vacuum.

This radiation shield permits maintaining operating temperature with a power input less than half that required for the unshielded tube, and with a temperature gradient of less than ten degrees along its length.

The water-cooled induction heating coil is wound using standard 4.75 mm copper tubing with hard-soldered joints. The center fed, parallel coil configuration minimizes high frequency excitation of the metal vapor or buffer gas in the furnace tube or vacuum chamber. The tank circuit of the induction heater oscillator is single ended. By applying the high potential to the center of the heating coil, discharge in the buffer gas or sample vapor is effectively eliminated. The induction heater used is a 5 KW portable unit operating at 450 kilocycles.

Notes:

1. Reference: Additional details may be found in Applied Optics, Volume 6, page 1299, August 1967. "A Modified King Furnace for Absorption Spectroscopy of Small Samples" by F. S. Tompkins and B. Ercoli, Chemistry Division of Argonne National Laboratory.
2. Inquiries concerning this innovation may be directed to:

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Reference: B68-10418

Source: F. S. Tompkins and B. Ercoli
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(ARG-10177)

Patent status:

Inquiries about obtaining rights for commercial use of this innovation may be made to:

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